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STABILITY-INDICATING ASSAY OF SULPHACETAMIDE IN THERMALLY DEGRADED SOLUTIONS

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ABSTRACT

A stability-indicating assay method for the determination of sulphacetamide in thermally degraded solutions is reported. The method is based on the difference in the absorption maxima of sulphacetamide and sulphanilamide at pH 4.0 and their simultaneous determination by a two component assay using the analytical wavelengths of 258nm and 268nm, the reproducibility of the method lies within ± 4%. It is rapid and specific for the assay of sulphacetamide in degraded opthalmic preparations.

Introduction

Sulphacetamide sodium in 5-30% concentrations is extensively used in ophthalmic preparations for various eye infections. Aqueous sulphacetamide solutions undergo hydrolysis to yield sulphanilamide on storage and heating (Clarke, 1965 and Ahmad, 1983). Sulphacetamide in thermally degraded solutions has been assayed colorimetrically after separation by TLC on silica gel (Anderson, 1966 and Davies et al., 1970). This method is time consuming and relatively less accurate.

In this work the development of a stability-indicating spectrophotometric method for the direct assay of sulphacetamide in presence of sulphanilamide is being reported. This method has been applied to the assay of sulphacetamide in ophthalmic preparations.

Materials and Method

Sulphacetamide and sulphanilamide were obtained from Sigma and BDH respectively. All the reagents used were of the analytical grade unless otherwise indicated.

10^{-4}M solutions of sulphacetamide and sulphanilamide were prepared at pH 4.0 (citrate buffer) and absorption spectra were determined on SP 800 Pye-Unicam Spectrophotometer. Synthetic mixtures containing different ratios of the two compounds were also prepared at the same pH. Analytical absorption measurements were made on SP 500 Pye-Unicam spectrophotometer.

10^{-3}M aqueous solutions (pH 7.0, phosphate buffer) were degraded in sealed ampoules at 90°C ± 1° in a thermostat oven. The ampoules were withdrawn at appropriate intervals of time and chilled to 2°C to stop the reaction. The sulphacetamide contents

were determined by a two component spectrophotometric assay using the wavelengths of 258nm and 268nm.

Table 1: Thermolysis of 10^{-3}M sulphacetamide solution at pH 7.0 (90°C) Concentrations of sulphacetamide and sulphanilamide.

Time (hrs.)	Sulphacetamide moles/1 x 10^4	Sulphanilamide moles/1 x 10^4	Total moles/1 x 10^4
0	10.000	0.000	10.000
50	9.440	0.311	9.751
100	9.016	0.739	9.755
150	8.462	1.262	9.724
200	7.741	1.869	9.610
250	7.586	1.936	9.522
300	7.261	2.239	9.500

Results and Discussion

The results of the assay of sulphacetamide in presence of sulphanilamide in thermally degraded solutions at 90° ± 1°C for 300 hours are given in Table-1 and the absorbance vs. time curves at the analytical wavelengths are shown in Fig. 1. The relatively large decrease in absorbance at 268nm as compared to that of 258nm indicates the gradual thermal transformation of sulphacetamide to sulphanilamide and affords spectroscopic evidence for the presence of later compound. Sulphanilamide was also detected in the degraded solutions by TLC on silica gel GF 254 (Rf 0.83, ethanol: methanol, (50:50) (Klein and Kho, 1962).

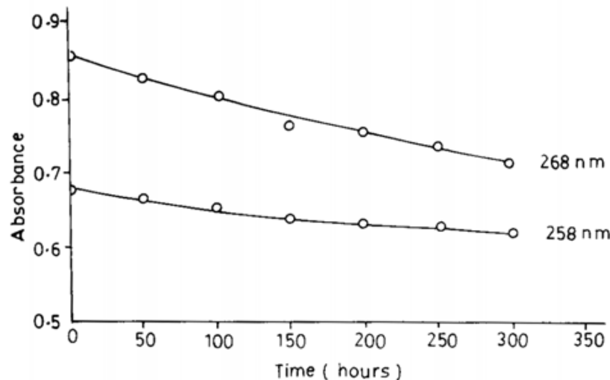

Fig. l: Themiolysis of 10°M Sulphaectamide solution at pH 20 (90°C). Absorbance measurements were made on diluted solutions (1:20) a pH 4.00.

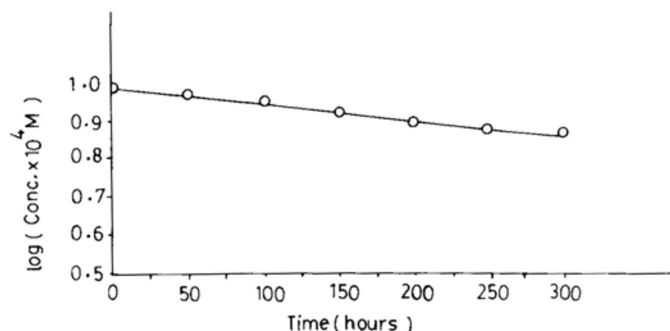

Fig. 2: First-order plot for the Nemmlysis of sulphacetamide solution at pH 7.0 (90°C).

The total molar balance for sulphacetamide and sulphanilamide Table-1 showed a gradual decreasing pattern and a value of 9.500 x 10^4M/1 at 300 hours suggests that some other minor product(s) may also been formed which could not be accounted for in this assay. However, when the analytical data for sulphacetamide were plotted on the log scale, a smooth straight line was obtained indicating the consistency of the calculated data (Fig. 2).

From the slope of the straight line the value of the first-order rate constant for the th-ennolysis of sulphacetamide at 90°C was determined as 1.07 x 10^{-3} hr

The analytical wavelengths of 268nm and 258nm used in the two component assay correspond to the absorption maxima of sulphacetamide and sulphanilamide at pH 4.0. At this maximum distinction is observed between the absorption maxima of the two compounds. The molar extinction coefficients used for calculations of the concentrations are as follows:

Sulphacetamide 268nm17100 258nm13500

Sulphanilamide 268nm 11600 258nm 15300

The reproducibility of the method was observed by the analysis of the binary mixtures of sulphacetamide and sulphanilamide at pH 4.0. It was found that the magnitude of the error is within ± 4% and hence the method can be conveniently used for the assay of sulphacetamide in ophthalmic preparations. It is specific, rapid and stability-indicating in the presence of sulphanilamide which is the main product in thermally degraded solutions.

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